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Microstructures after Processing by Aging and ECAP for Al-Mg_2Si Alloys Containing Excess Si or Mg

Kazuko Fujita[1,a], Takeshi Fujita[2,b], Keiichiro Oh-ishi[3,c], Kenji Kaneko[1,d], Zenji Horita[1,e] and Terence G. Langdon[4,f]

[1]Department of Materials Science and Engineering, Faculty of Engineering
Kyushu University, Fukuoka 812-8581, Japan

[2]Center for Solid State Science, Arizona State University, Tempe, AZ 85287-1504, USA

[3]Department of Mechanical Engineering, Naval Postgraduate School,
700 Dyer Road, Monterey, CA 93943-5146, USA

[4]Departments of Aerospace & Mechanical Engineering and Materials Science
University of Southern California, Los Angeles, CA 90089-1453, USA

[a]fujita_k@zaiko6.zaiko.kyushu-u.ac.jp, [b]Takeshi.Fujita@asu.edu, [c] masakei1@yahoo.co.jp,
[d]kaneko@zaiko.kyushu-u.ac.jp, [e] horita@zaiko.kyushu-u.ac.jp, [c]langdon@usc.edu,

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Abstract. In this study, severe plastic straining through equal-channel angular pressing (ECAP) is imposed on age-hardenable Al-Mg-Si alloys having two different compositions of excess Si or Mg in the Al-Mg_2Si pusedo-binary system. Thereafter, the alloys are subjected to aging and the microstructures are examined using transmission electron microscopy. It is shown that the precipitation behavior is significantly changed through application of ECAP.

Introduction

Equal-channel angular pressing (ECAP) is a processing technique to introduce intense strain into metallic materials [1] and thus not only refines grain sizes to the submicrometer range [2] but also changes morphology of second phase particles [2,3]. Application of the ECAP process for age-hardening alloys showed that the precipitates are deformed or fragmented by strong shear introduced by the ECAP process and they may dissolve into the matrix [2-4]. It was also shown that the aging behavior is significantly changed when aging is performed on the ECAP samples [3,4].

Aluminum alloys containing dilute amounts of Mg and Si are the typical alloy system exhibiting age hardening. Various types of particles are formed during aging process but they depend not only on the aging time and temperature but also on the relative amount of Mg to Si [5]. An earlier study using a balanced composition in the Al-Mg_2Si pusedo-binary system showed [4] that the metastable rod-shaped β' particles were fragmented into round-shaped particles through ECAP and some fraction was dissolved into the matrix. Aging after the dissolution of the particles led to a formation of cube-shaped particles whereas such particles were never observed in the balanced composition but only in the composition with excess Mg [6]. It was also shown [4] that the aging effect was accelerated by severe straining through ECAP or ECAP before aging led to a formation of a new phase. For a balanced composition, the metastable rod-shaped β' particles formed after much shorter annealing time than the normal. There was also a formation of Si particles when aging was continued for prolonged time.

In this study, ECAP is conducted on two different compositions with excess Mg and excess Si. Precipitation behavior associated with aging after ECAP is examined using transmission electron microscopy.

Experimental Procedures

Alloys having two different compositions were prepared through melting and casting procedure: one has excess Mg with a composition of Al-1.0wt%Mg_2Si-0.4wt%Mg and the other contains excess Si with a composition of Al-0.7wt%Mg_2Si-0.7wt%Si. Ingots of the alloys were homogenized at 848 K for 24 hours and swaged into rods having diameters of 10 mm. The rods were cut into lengths of 60 mm and each was solution-treated at 848 K for 1 hour. These alloys were aged at 473 K for up to 30 hours so that the metastable β' phase formed in the alloys. ECAP was then undertaken on such aged alloys at room temperature for 8 passes through route B_C using a die having a channel angle of 90^o and aging was further conducted at 473 K for 1 hour and 30 hours (aging + ECAP + aging). ECAP was also conducted directly on the solution-treated alloys at room temperature for up to 8 passes through route B_C using the same die. Aging was then undertaken at 473 K for 1 hour and 30 hours (ECAP + aging).

A Hitachi H-8100 transmission electron microscope (TEM) was used for observations of bright field images and dark field images. Lattice images were observed using a JEM2010FEF energy-filtered transmission electron microscope. The latter was also operated in scanning mode (STEM) and X-ray mapping was undertaken using the Al-K_α, Mg-K_α and Si-K_α lines. Thin specimens for such electron microscopy were prepared using a twin-jet electro-polishing technique in a solution of 20%$HClO_4$, 10%$C_3H_8O_3$ and 70%C_2H_5OH.

Results and Discussion

Aging + ECAP. Figure 1 shows (a) a TEM bright field image and (b) a dark field image including (c) an SAED pattern of the sample containing excess Si after annealing for 1 hour at 473 K. The dark filed image was taken using a diffraction spot indicated by the arrow. Many rod-like precipitates are visible which are elongated along the <100>$_m$ direction of the matrix. A lattice image of a precipitate is shown in Fig.2 (a) and a diffractogram taken from the precipitate is in Fig.2(b). Close inspection of the image and diffractogram reveals that this particle is a type A

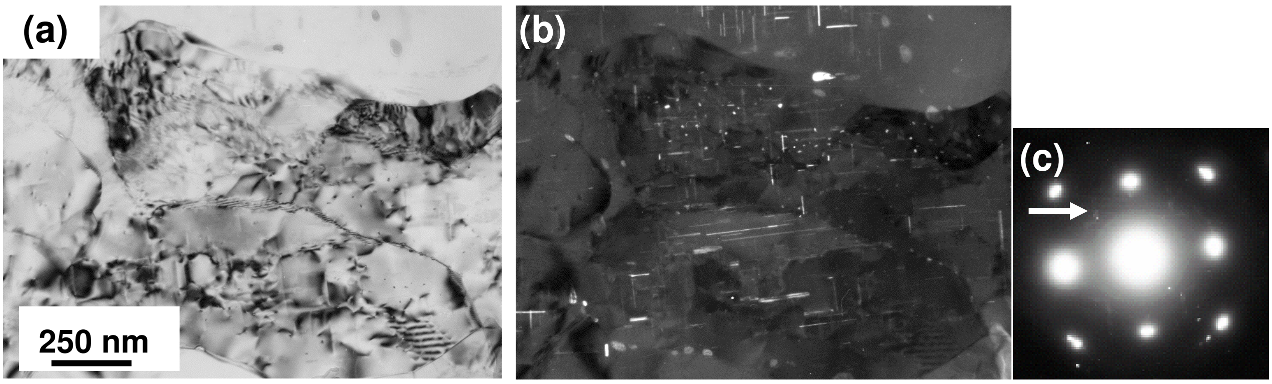

Fig. 1 (a) TEM bright field image and (b) dark field image including (c) SAED pattern of the sample containing excess Si after annealing for 1 hour at 473 K

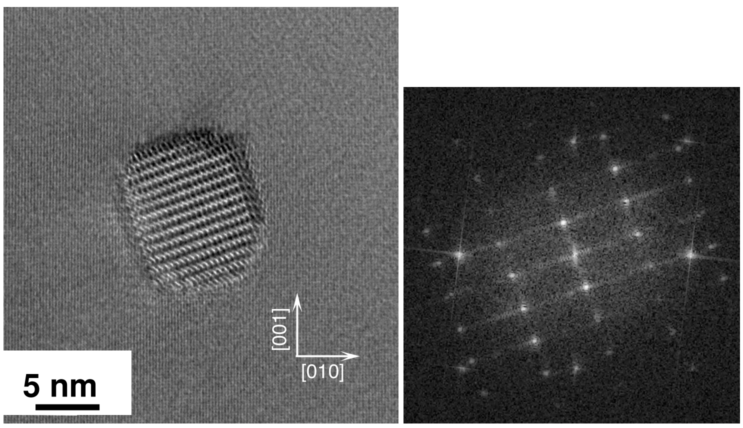

Fig. 2 (a) Lattice image of type A precipitate and (b) diffractogram taken from the precipitate

precipitate. Matsuda and Ikeno reported [5] that the formation of type A precipitates occurred in a solution-treated sample after prolonged aging such as for 1000 hour at 473 K. Thus, the aging for 1 hour for the present ECAP sample is very short and it is considered that the aging was accelerated by severe plastic strain introduced by the ECAP process. This result is consistent with an earlier observation [4] that precipitation process proceeds at a faster rate than that in the normal strain-free condition.

Figure 3 shows (a) a STEM image and (b)-(d) X-ray mappings of the corresponding area taken with the Al K_α, Mg K_α and Si K_α lines for the sample containing excess Mg. The sample was aged for 30 hours at 473 K. The X-ray analysis reveals that the particle indicated by the arrow in Fig.3 (a) is rich in Si. This is also confirmed with an X-ray spectrum shown in Fig.4. There is no peak of Mg and it is concluded that a Si phase formed in the sample despite the composition which is excess of Mg. Although an oxygen peak is present, this must be due to an oxide layer formed during electro-polishing for electron microscopy. The presence of the Si particle should be attributed to the introduction of severe plastic strain through the ECAP process. The presence of Si particles was also observed in an earlier study of a balanced Al-0.9%Mg2Si alloy after ECAP process [4].

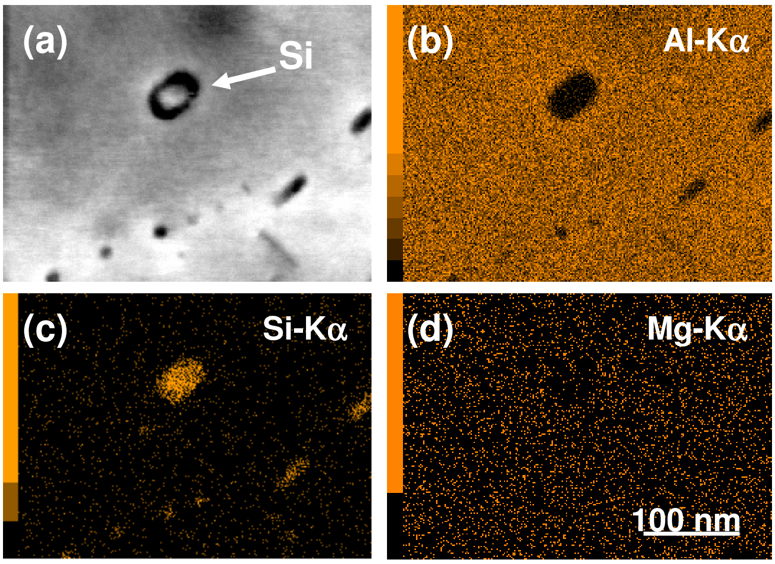

Fig. 3 (a) STEM image and (b)-(d) X-ray mappings using Al K_α, Si K_α and Mg K_α lines for excess-Mg. alloy

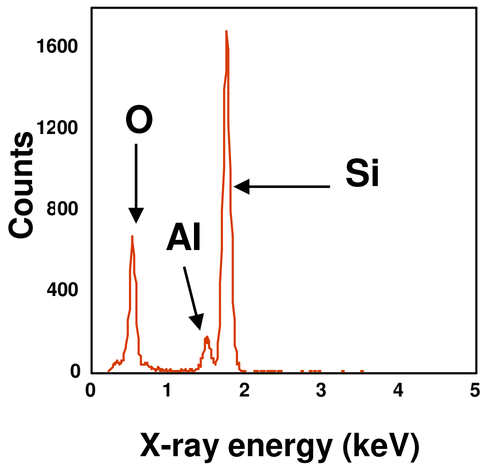

Fig. 4 X-ray spectrum from particle indicated by arrow in Fig.3 (a)

Aging + ECAP + Aging. The samples subjected to aging and ECAP were further aged for 30 hours at 473 K. Figures 5 are dark field images of the samples containing (a) excess Si and (b) excess Mg, respectively. The SAED patterns are included in both figures and they confirm the presence of cube-shaped particles due to the arrays of extra spots. The dark field images were taken with the diffracted beams indicated by the arrows in the SAED patterns. The cube–shaped particles are visible in both alloys but the size and distribution are smaller and finer in the alloys with excess Mg than with excess Si. Lattice image observation showed that the cube–shaped particles are present even after aging for 1 hour in both alloys. An example of the image including diffractogram is shown in Fig.6.

The formation of the cube-shaped particles was often observed in the excess Mg alloy after solid solution treatment and subsequent aging. However, it should be emphasized that the cube-shaped particles has also formed in the excess Si alloy after treatments of aging, ECAP and aging for the present study. It is considered that the severe plastic straining through the ECAP process has played a significant role for the formation of the cube-shaped particles in the excess Si alloy. It should be noted that the cube-shaped particles neither formed in the excess Mg alloy nor in the excess Si alloy when aging is attempted for the ECAP sample without prior aging but directly after solution treatment. Further study is required to clarify such complication.

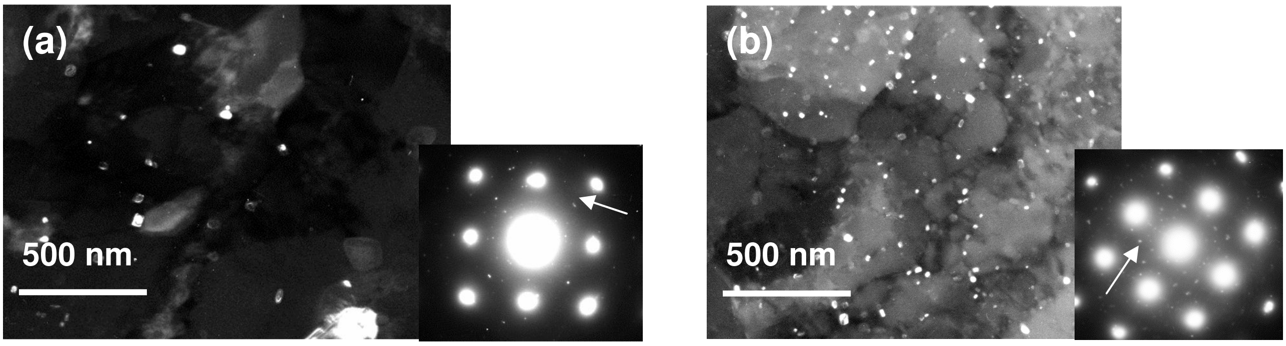

Fig. 5 Dark field images of (a) excess- Si alloy and (b) excess-Mg alloy aged for 30 hours at 473 K following aging and ECAP.

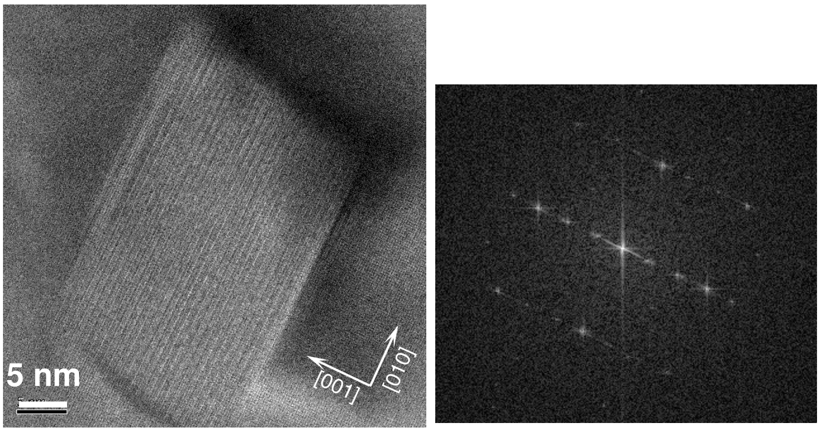

Fig.6 Lattice image and diffractogram of cube–shaped particle after aging for 1 hour in excess-Si alloy.

Conclusions

(1) Precipitates of A type were observed in the excess-Si alloy after short time aging as 1 hour when aging was performed on the ECAP sample.
(2) Precipitates of Si phase were formed in the excess-Mg alloys after aging of the ECAP sample.
(3) Cube-shaped particles were formed not only in the excess-Mg alloy but also in the excess-Si alloy after aging of the samples subjected to aging and ECAP. However, they were observed in neither alloy when aging was performed on the ECAP sample without prior aging.

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